

# **Fabrication and Characterization of Multi-Walled Carbon Nanotube (MWCNT) and Ni-coated Multi-Walled Carbon Nanotube (Ni-MWCNT) Repair Patches for Carbon Fiber Reinforced Composite Systems**

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Multi-walled carbon nanotube (MWCNT)/epoxy and nickel-coated multi-walled carbon nanotube (Ni-MWCNT)/epoxy systems were fabricated into carbon fiber composite repair patches via vacuum resin infusion. Two 4 ply patches were manufactured with fiber orientations of [90/ 90/ 45 90] and [0/ 90/ +45/ -45]. Prior to resin infusion, the MWCNT/Epoxy system and Ni-MWCNT/epoxy systems were optimized for dispersion quality. Scanning electron microscopy (SEM) and optical microscopy (OM) were used to determine the presence of carbon nanotubes and assess dispersion quality. Decomposition temperatures were determined via thermogravimetric analysis (TGA). SEM and TGA were also used to evaluate the composite repair patches.

## *Introduction*

Composites are materials that consist of two or more different types of materials that come together to create something with properties each material could not accomplish of its own [6]. The constituents of carbon fiber reinforced composites are a reinforcing phase of carbon fiber and a matrix phase of epoxy resin [4]. The fibers are embedded in the epoxy resin and allowed to cure. Each phase has its own physical and chemical properties; combined they achieve properties that each could not do alone [8]. A strong bond must exist between the fibers and the resin; weak bonding will cause delamination in the composite structure. The strength of the composite depends on the orientation of the fibers[4].

The advantages of using composites are that they are light weight, strong, easily assembled, save energy, and can be molded into different shapes [1]. They have generally been used in the automotive, aerospace, and sports industries. In the future, they will be used for alternative energy, construction, and oil exploration [13].

Carbon fiber was first used by Thomas Edison as filaments for light bulbs. He used carbon fibers because of their tolerance to heat which made them ideal for conducting electricity. Edison's carbon fibers were made out of cellulose-based materials (cotton and bamboo) that were carbonized [5]. Today, the most common precursor is polyacrylonitrile (PAN); rayon and petroleum also are often used. The process used to make the fabric from the precursors is called carbonization. Carbonization is when the precursor is drawn into long strands and then heated without oxygen so that the fiber cannot burn. The atoms of the fiber vibrate until almost all of the non-carbon atoms are gone. Once this is completed a fiber of long, tightly inter-locked chain of carbon atoms are produced. These long thin fibers are about 0.005-0.010 mm (0.0002-0.0004 in) in diameter. The carbon atoms are bonded in a microscopic crystal alignment. The crystal alignment is what makes the fibers strong [13]. The fibers are what give the composite its strength because of their high specific strength and stiffness and do not degrade with temperature. Although they are strong, they do not have structural value which can cause them to degrade chemically when exposed to the atmosphere [4].

The epoxy resin is a thermoset polymer resin. The material contains a collection of a large number of polymer molecules of similar chemical structure. These molecules are chemically joined together by cross-links that form a rigid three-dimensional structure. Thermoset polymer resins cannot be reshaped and reformed once cured

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with the carbon fiber. Epoxy requires a curing agent (catalyst) to allow the thermoset polymers to cure. The cross-linking occurs when the polymer goes through a chemical reaction.[4] The Epoxy is used to protect the fibers from environmental and chemical damage. It also keeps the carbon fibers in the desired orientation. When the epoxy and fibers are bonded, the applied load (stress) is transferred between the fibers [9].

Multi-walled carbon nanotubes (MWCNT) are a coaxial assembly of 2 – 50 graphite sheets. Their diameter can range from 5 – 50 nm [10]. The graphite sheets are attracted to each other by Van de Waals forces. The distance between the graphite sheets is approximately 0.340 nm. Scientists believe that composites can be designed with good mechanical, thermal, and electronic properties[11]. Carbon nanotubes were first discovered by Iijima, a Japanese scientist. The electronic transport that occurs in the nanotubes is ballistic in metallic nanotubes which cause them to conduct electricity with minimal resistance. Many nanotubes are used for structural application and medicine. Combining nanotubes with the polymer matrix increases that material's mechanical strength, conductivity, fracture toughness, and electromagnetic shielding properties[8]. MWCNT can be coated with a metal to give them conductive properties and electromagnetic shielding. Ni-coated Multi-walled carbon nanotubes (Ni-MWCNT) are effective because nickel is conductive, magnetic, and corrosion resistant. It increases the tensile strength and damping properties[3].

Since the incorporation of composite components into aircraft, aerospace, and automotive parts, there has been a need to continually improve upon the repair procedures and techniques of these parts. Several repair types exist depending on the damage and duration that part was in service. One such repair is a bonded repair in which the carbon fiber is cured and then adhesively bonded to the structure. The advantages of repair patches are that no holes need to be drilled into the part which reduces stress concentrations and they can be readily formed to any shape which allows for the ability to repair irregularly shaped parts. Pre-cured repair patches can be used for temporary and permanent repairs[2].

For this experiment the MWCNT and Ni-MWCNT were analyzed in the epoxy system for thermal stability and dispersion. Thermogravimetric analysis (TGA) was used to test the thermal stability by measuring the decomposition temperature of the MWCNT/epoxy system and the Ni-MWCNT/epoxy system. The dispersion was evaluated by SEM and OM. Once the initial analysis was done, the carbon fiber was vacuum resin infused and evaluated via micro hardness, OM, and TGA.

### *Experimental*

#### **MWCNT and Ni-MWCNT Preparation:**

The MWCNTs and Ni-MWCNTs were received from cheaptubes.com (112 Mercury Drive, Brattleboro, VT 05301 USA). The carbon nanotubes were manufactured by Catalytic Chemical Vapor Deposition (CCVD). The Ni-MWCNTs were plated with nickel after the CCVD process. The MWCNTs had an outer diameter that was less than 8 nm and the Ni-MWCNT's outer diameter was 10-20 nm. The length of the carbon nanotubes was 10-30  $\mu\text{m}$ .

#### **SEM Sample Preparation of MWCNT/Ni-MWCNT nanoparticles:**

A small amount of MWCNTs and Ni-MWCNTs was added to different glass vials. Isopropanol was added to each of the vials. The vials were sonicated in a Branson Ultrasonicator for five minutes. Two petri dishes were obtained and covered in foil. A three millimeter grid was put on each petri dish. The samples were put on the grids using glass droppers. The grids were then allowed to dry. Once dry the grids were coated with carbon in the JOEL/JEE-420 Vacuum Evaporator for SEM imaging on a JEOL-JSM 7500F.

#### **Curing MWCNT/Epoxy System and Ni-MWCNT/Epoxy System: Method A**

The resin used for this experiment was Aeropoxy which was purchased from Aircraft Spruce; a two part epoxy system that consists of resin and hardener. The best mixing ratio of resin to hardener was 100:27 (3:1). The amount of the MWCNTs or Ni-MWCNTs used was 0.5% of the total weight of the epoxy system. The hardener was weighed to 49.005 g in a glass beaker. 1.153 g of MWCNTs were added to the hardener and mixed with a Servodyne Mixer (Figure 1) for 30 minutes at 150 rpm. After mixing, the hardener and MWCNT were added to 181.5 g of resin. This was mixed with the mixer for 10 minutes at 200 rpm. Once the systems were done mixing, they were added to aluminum pans lined with parafilm which was used as release film. The samples were allowed to cure overnight. The procedure was repeated for the Ni-MWCNTs. The control/neat epoxy consisted of the epoxy resin and hardener only.



Figure 1: Servodyne Mixer

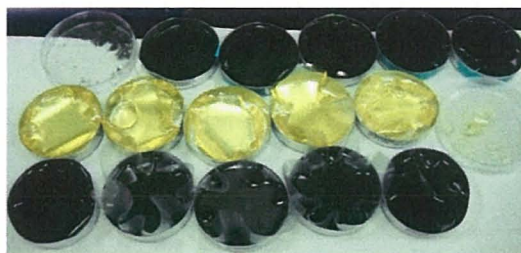


Figure 2: Cured Method A Epoxy and Carbon Nanotube Samples

#### SEM Sample preparation of Method A Samples:

A Struers 36TRE cut off wheel (Figure 3) was used to section the samples. One piece of the sample was hit with a hammer to obtain shard samples. It was determined that the shard samples were easier to evaluate in the SEM for dispersion of the MWCNTs and Ni-MWCNTs in the epoxy systems. The SEM procedure involved mounting the shard samples on carbon tape and then gold-coating.



Figure 3: Struers Cut off Wheel

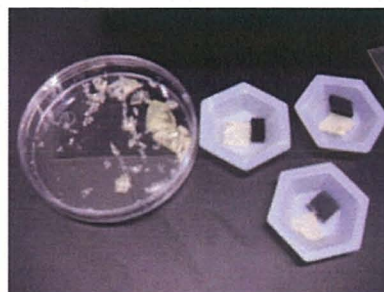


Figure 4: Shard and Cut Cured Samples

#### TGA of Method A Samples:

A TA Instruments Q5000 TGA (Figure 5) was used to obtain decomposition data. Shard samples were heated to 800°C in nitrogen purge at a rate of 20°C/min.



Figure 5: TGA Q5000

#### Curing MWCNT/Epoxy System and Ni-MWCNT/Epoxy System: Method B

To further optimize the dispersion of the carbon nanotubes another method was evaluated. The hardener was weighed to 19.17g and mixed with 0.451g of MWCNTs using a Banson Ultrasonic Processor sonicating probe (Figure 6) for one hour at 50% amplitude. An ice bath was used during the mixing process to control the heat and to keep the epoxy hardener from solidifying too quickly. After one hour of mixing, the MWCNT/hardener mixture was added to 71g of resin and stirred using a wooden stirrer. The MWCNT/epoxy system mixture was transferred to

aluminum pans lined with parafilm (release film). The samples were allowed to cure overnight. The same procedure was repeated for the Ni-MWCNTs. The control/neat epoxy consisted of epoxy resin and hardener.



Figure 6: Probe Sonicator in Ice Bath

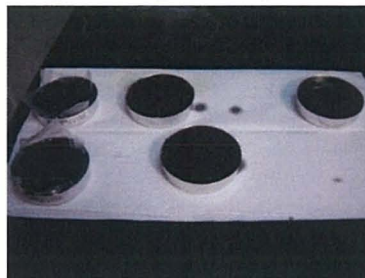


Figure 7: Cured Method B Epoxy and Carbon Nanotube Samples

#### OM Sample Preparation of Method B Samples:

The cured samples were sectioned into pieces using a hacksaw. The pieces were polished on a Struers Grinder/Polisher using 120, 400, and 800 grit paper. The diamond paste used to polish them was 9, 6, and 3 microns. The final polish was done with a silica polish slurry. The polished end of the samples were sliced with a razor blade and put under the optical microscope for imaging.



Figure 8: Cured Sample cut with Hacksaw

#### TGA of Method B Sample Shavings:

Small Shavings were milled (figure 9) from the cured samples for TGA. The samples were run at 20°C/min to 800°C in a nitrogen purge.

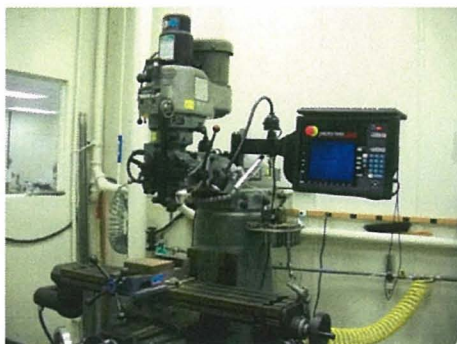


Figure 9: Milling Machine



Figure 10: Sample Shavings

#### TGA of Method B Shard Samples:

The sample preparation was modified because there was a possibility that the heat from the milling machine was affecting the thermal properties of the epoxy system. One piece of the puck sample was hit with a hammer to obtain shard samples. The samples were run at 20°C/min to 600°C.





Figure 11: Shard Samples

### Dry Run Infusion:

To test the infusion of the epoxy system with MWCNTs and Ni-MWCNTs and determine an estimated amount of epoxy system to infuse, a dry run was performed. One ply of 3K (3000 filament/thread) carbon fiber was used and the lay-up schedule consisted of plastic, peel ply, fiber, peel ply, flow medium, intake and vacuum manifolds, and plastic bag. The plastic on the bottom is used so that the fiber does not stick to the table when curing. Peel ply takes up the excess resin so that the minimum amount of resin is in the fibers. Flow medium and the manifolds are what help the resin flow better and faster through the fabric. The plastic bag on top is tacky taped to the table so that the infusion could be done under vacuum with a pump. The total amount of epoxy resin and hardener that was infused was 2 oz. Before infusion, 12.05g of hardener was mixed with 0.284g of MWCNTs with the probe sonicator for one hour at 50% amplitude. The hardener and MWCNTs were added to 44.65g of resin and mixed with a wooden stirrer. The epoxy system with the MWCNTs was allowed to sit for ten minutes to let all the air bubbles travel to the surface. The epoxy system and MWCNTs was infused into the fiber through a plastic tube. Once the infusion was done, the tube was clamped closed and the vacuum remained on until the fiber cured.

### Infusion of Repair Patches:

The lay-up for the infusion was plastic, carbon fibers; peel ply, flow medium, intake and vacuum manifolds, and plastic bag. Four ply 3K carbon was infused. Each fiber was half an inch smaller, which caused a quarter of an inch over-lay. Two different patches were infused. The fibers were stacked on top of each other in the specified order and weighed before being infused. The amount of epoxy system used to infuse was reduced to minimize waste. The probe sonicator was used to mix 9.95g of hardener and .234g of MWCNTs for 20 minutes at 30% amplitude in an ice bath. Following the mixing, the hardener and MWCNTs were added to 36.9g of resin and mixed with a wooden stirrer. The epoxy system was allowed to sit for ten minutes to let all the air bubbles travel to the surface. The fibers were infused under vacuum and once the epoxy completely wet the fibers, the plastic tube was clamped closed and the vacuum remained off until the fibers fully cured. The lay-up was left overnight, and then the cured fibers were weighed again. This process was repeated with the Ni-MWCNT/epoxy system and the control/neat epoxy.

Ply	Patch 1	Patch 2
1	90°/2.5 x 4.5	0°/2.5 x 4.5
2	90°/3 x 5	90°/3 x 5
3	45°/3.5 x 5.5	+45°/3.5 x 5.5
4	90°/4 x 6	-45°/4 x 6



Figure 12: Repair Patches Infusion Set Up

#### Micro Hardness Testing Sample Preparation of Repair Patches: Top Side

A strip was cut out the middle of each patch and a small piece was cut from the center of each strip. The resin rich side of the patch was mounted into a puck using Kold Mount, which is a two part specimen mounting material. The patch pieces were set on the bottom of a plastic cup with the resin rich side up. The mounting material was poured on top of the samples and allowed to cure for 30 minutes. The sample puck was taken out of the plastic cup and polished to get the mounting material off the top of the patch pieces. The micro hardness testing was done with a Buehler Micromet II Digital Micro Hardness Tester (Figure 15). A Vickers diamond indenter was used at 50g, 100g, 300g, and 500 g loads.



Figure 13: Mounting Material

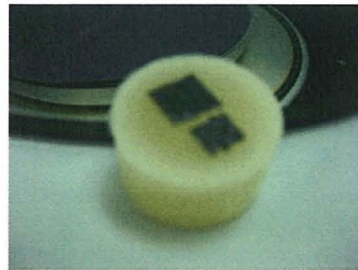


Figure 14: Mounted Patch Top Side Samples



Figure 15: Micromet II Digital Micro Hardness Tester

#### Micro Hardness Testing Sample Preparation of Repair Patches: Cross Section

Another sample piece was cut from each patch strip. The plastic cup was filled about one-fourth of the way with the mounting material. The samples were set up straight in the plastic cup with the cross section touching the bottom. Once all the samples were in the cup, the cup was filled to the top with mounting material and allowed to cure for 30 minutes. The puck was taken out of the plastic cup and polished so that the samples would be exposed for testing. The samples were then tested for their micro hardness with the Vickers diamond indenter at 50g, 100g, 300g, and 500g loads.



Figure 16: Mounted Patch Cross Section Samples

#### OM of Repair Patches:

A sample piece was cut from the strip of each patch. Each piece was put in a multi-clip that is used to hold samples when mounting. The samples were mounted in a phenolic hot mounting resin with carbon fiber. The sample pucks were mounted in a Struers Labo Press-3 (Figure 17) at 180°C at 30 kN for six minutes. When the samples were taken out of the mounting press they were polished with a Struers Tegra Pol-11 (Figure 18) with 120 and 600 grit paper, then nine micron particle paste. After polishing the samples were imaged by OM.



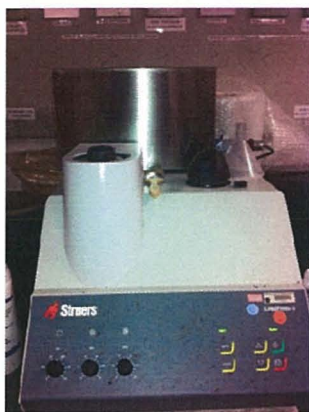


Figure 17: Struers Labo Press-3



Figure 18: Struers Tegra Pol-11

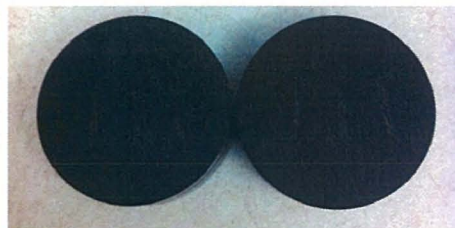


Figure 19: Mounted Patch Samples

### *Results and Discussion*

#### **SEM Analysis of MWCNT/ Ni-MWCNT**

Figures 20 and 21 show that the MWCNTs and Ni-MWCNTs are bundled together because of Van de Waals intermolecular forces. As shown in the images, the diameters of the tubes are consistent with vendor specifications.

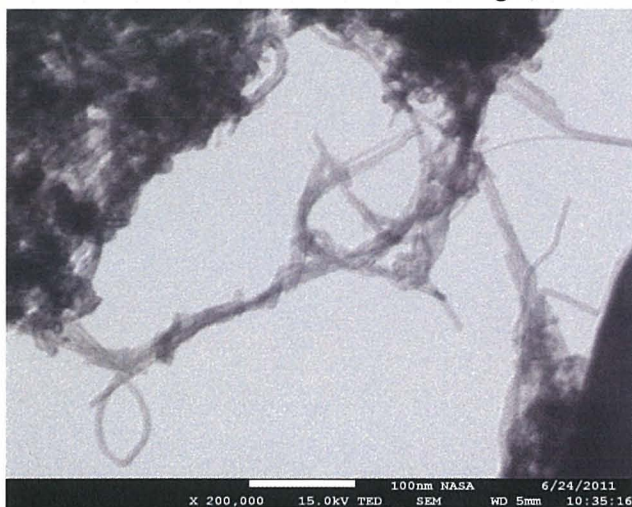


Figure 20: SEM image of MWCNT



Figure 21: SEM image of Ni-MWCNT

#### **Method A Analysis:**

##### **SEM analysis**

SEM revealed the presence of MWCNTs and Ni-MWCNTs in the epoxy system in figures 22, 23, and 24. SEM was not able to assess the quality of dispersion, but visible inspection revealed large agglomerates in the MWCNT/epoxy system and the Ni-MWCNT/epoxy system.

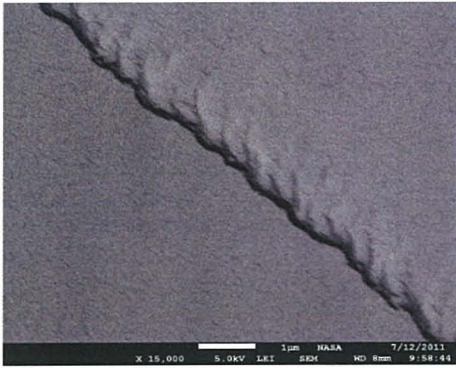


Figure 22: Control/Neat Epoxy Method A

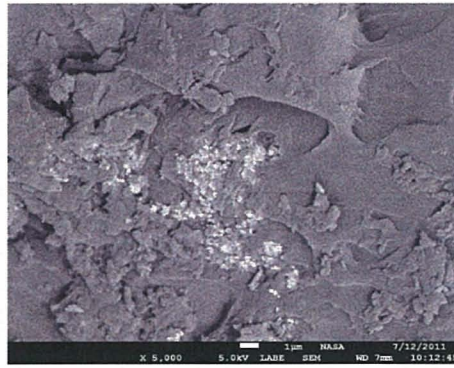


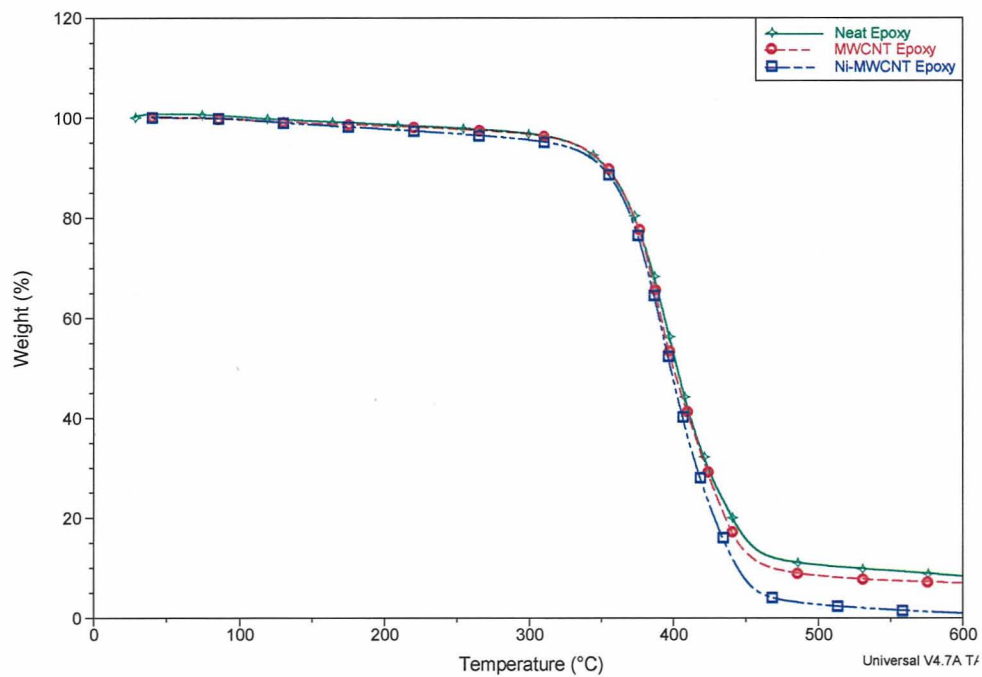
Figure 23: Ni-MWCNT in Epoxy System Method A



Figure 24: MWCNT in Epoxy System Method A

### TGA Data of Method A:

Sample Identification	Temperature (°C)
Control/Neat Epoxy	363
MWCNT/Epoxy System	362
Ni-MWCNT/Epoxy System	363



The TGA data shows that the incorporation of the carbon nanotubes did not alter the thermal stability of the polymer.

### Method B Analysis:

#### OM analysis

OM analysis of method B samples showed that the MWCNTs were well dispersed in figure 26 , however the Ni-MWCNT appeared to be bundled together in large agglomerates shown in figure 27.



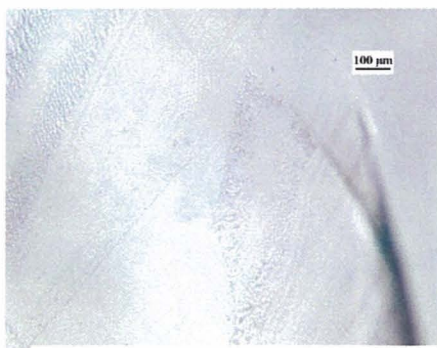


Figure 25: Control/Neat Epoxy Method B



Figure 26: MWCNT in Epoxy System Method B

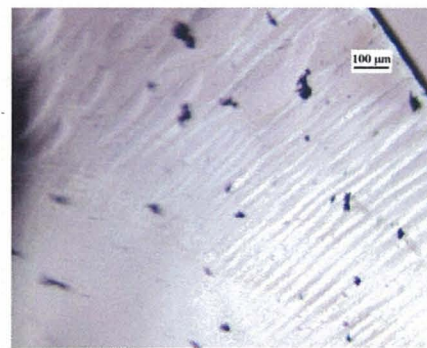
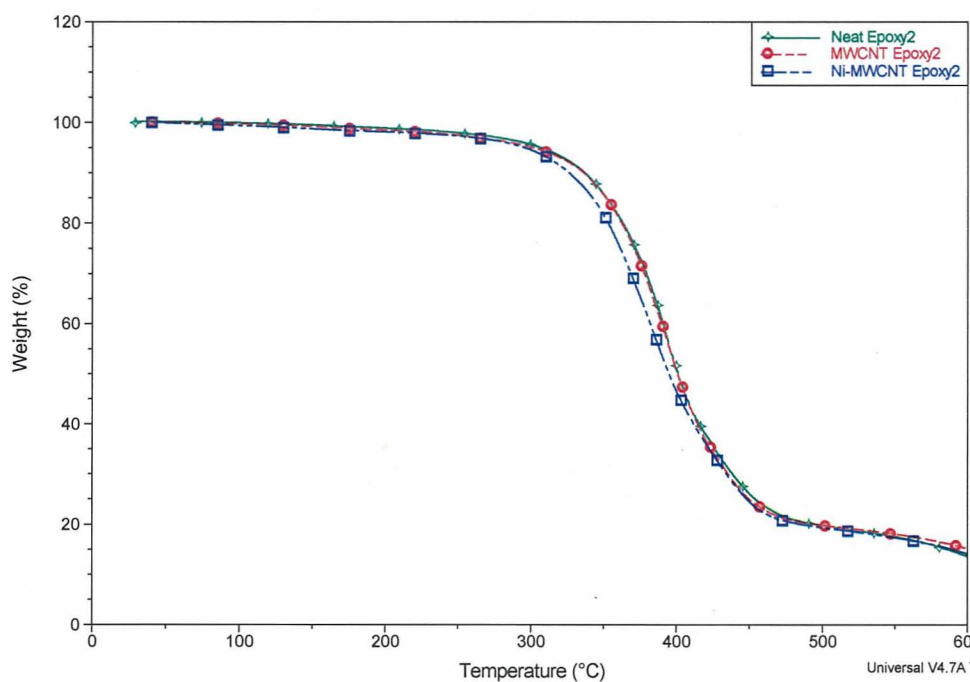


Figure 27: Ni-MWCNT in Epoxy System Method B

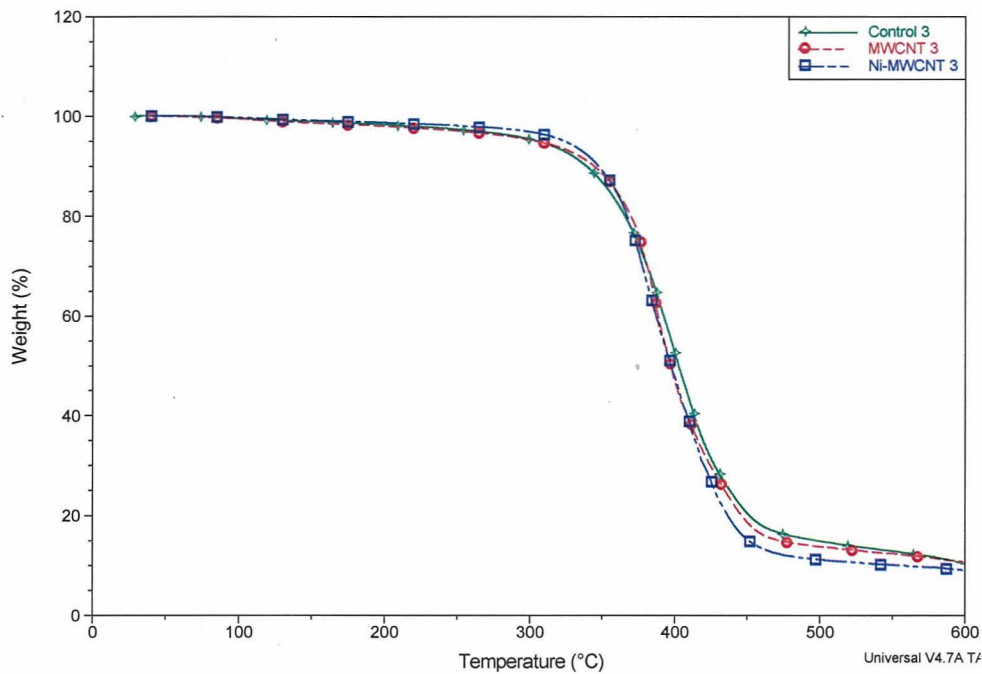
### TGA Data of Method B Milled Samples:

Sample Identification	Temperature (°C)
Control/Neat Epoxy	352
MWCNT/Epoxy System	347
Ni-MWCNT/Epoxy System	334



### TGA of Method B Shard Samples:

Sample Identification	Temperature (°C)
Control/Neat Epoxy	354
MWCNT/Epoxy System	363
Ni-MWCNT/Epoxy System	352



The TGA data shows that there was a decrease in the decomposition temperature when the probe sonicator was implemented, however the milling of the sample did not affect the temperature. The direct heat from the sonicator could have degraded the hardener and according to the MSDS, the hardener is unstable at elevated temperatures. As for the MWCNT/epoxy system, it appears that the decomposition temperature stayed constant when sonicated which could mean that the MWCNTs were stabilizing the hardener while sonication, but the milling of the sample could have possibly degraded the epoxy system and decreased its thermal stability. The Ni-MWCNT's decomposition temperature was first decreased by the sonicator and decreased more by the milling of the sample. It's apparent that the hardener was first destabilized by the heat from the sonicator and the epoxy system was degraded from the milling machine.

#### Micro Hardness Test of Repair Patches:

The micro hardness test was inconclusive for both the top side and cross section of the patch sample. The indentation from the Vicker's test could not be seen on the surface of either of the samples at each of the different loads. This most likely happened because of the elasticity of the fibers in the patch. It is possible that the samples were not hard enough after being indented with the Vickers test, the fibers would immediately move back into place not leaving an indentation or something too small to measure. Optimization of the micro hardness method will be continued. According to literature micro hardness has been successfully performed on carbon epoxy systems.

#### OM of Repair Patches:

OM shows indicated epoxy system regions (figure 25) and the fibers (figure 29), but the nanotubes could not be seen. OM revealed that there were some voids in certain regions of the patch, like in figure 26. Some damage to the patch is visible, but that is most likely due to the cutting of the patch.

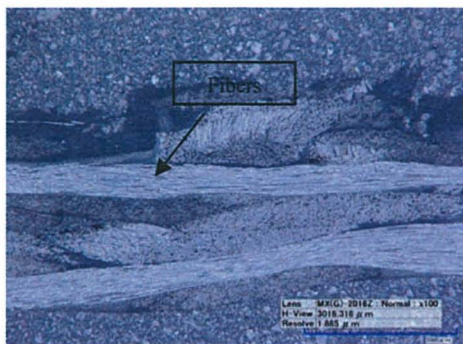


Figure 28: Patch 1: Control/Neat Epoxy in Carbon Fiber  
Kennedy Space Center

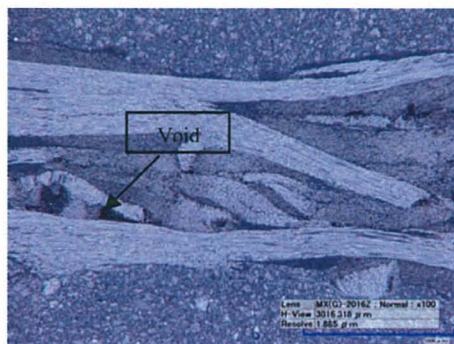


Figure 29: Patch 1: MWCNT/Epoxy System in Carbon Fiber  
10

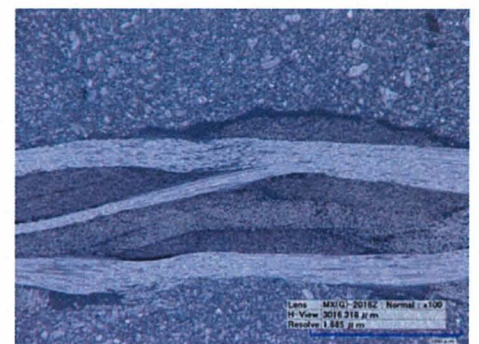


Figure 30: Ni-MWCNT/Epoxy System in Carbon Fiber  
Summer 2011



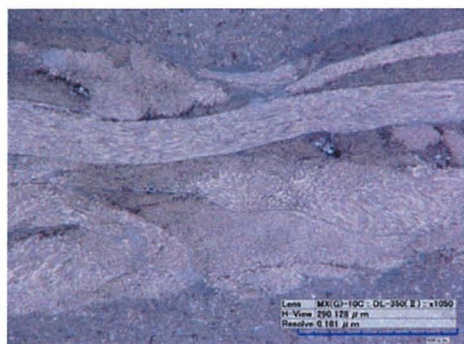


Figure 31: Patch 2: Control/Neat Epoxy in Carbon Fiber

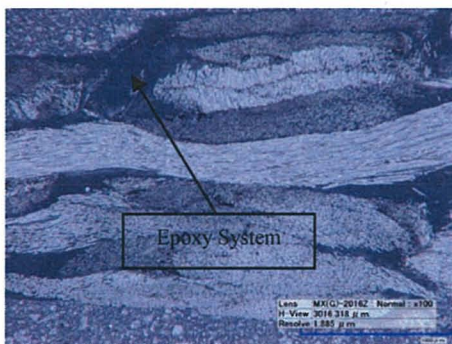


Figure 32: Patch 2: MWCNT/Epoxy System in Carbon Fiber

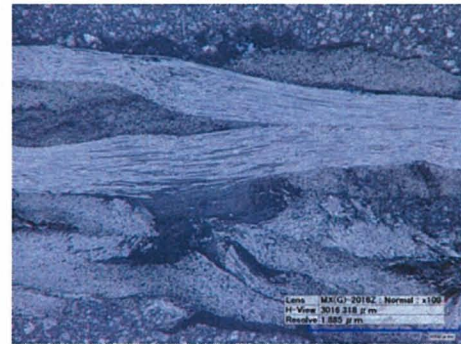


Figure 33: Patch 2: Ni-MWCNT/Epoxy System in Carbon Fiber

#### TGA data of Repair Patches:

Patch Identification	Control in CF (°C)	MWCNT/Epoxy System in CF (°C)	Ni-MWCNT/Epoxy System in CF (°C)
1	354	359	350
2	354	352	344

Although at this time a conclusive explanation of the decomposition mechanism cannot be completely explained, it is evident that the Ni-MWCNT/epoxy system in carbon fiber exhibited a lower decomposition onset temperature than the control or the MWCNT/epoxy system in carbon fiber. The system will be further optimized and further testing will be conducted to try and understand the system.

#### Conclusions

The probe sonicator dispersed the MWCNTs in the hardener better than the Servodyne mixer; however the Ni-MWCNTs were still bundled together after applying Method A and Method B. The MWCNTs and the Ni-MWCNTs did not modify the thermal stability of the epoxy system, but the use of the probe sonicator appears to have decreased the decomposition temperature of the control/neat epoxy by destabilizing the hardener. The MWCNTs seemed to have protected the hardener during sonication, but the milling machine apparently degraded the epoxy system. It appears that after sonication of the Ni-MWCNT/hardener, there was a decrease in the decomposition temperature, and after milling the cured sample the decomposition temperature decreased more. The Ni-MWCNT/epoxy system in Carbon fiber had the lowest decomposition temperature for both patches, but no further information could be concluded from TGA data of the patches. The OM revealed that there were some voids in the patch due to manufacturing issues or the infusion process and damages from sample preparation.

#### Future Work

There are multiple avenues to optimize our research. These will include further mechanical testing, composite manufacture and repair techniques, as well as thermal analysis. Some of the key aspect of this work will include the following:

1. Fabrication modifications to composite repair patches.
2. Optimize dispersion techniques for Ni-MWCNTs.
3. High-Res TGA which allows for separation of overlapping or poorly defined weight loss events. [12]
4. Decrease voids in repair patches by changing infusion process or using another resin system.
5. Use a better cutting technique for sample preparation of the patches.
6. Obtain better understanding of repair patches by doing an actual repair.

### *Acknowledgments*

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# FABRICATION AND CHARACTERIZATION OF MWCNT AND NI-MWCNT REPAIR PATCH FOR CARBON FIBER REINFORCED COMPOSITE SYSTEMS

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NASA USRP Summer 2011

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Materials Science Division

NE-L



# Purpose

- ▣ Analyze MWCNT and Ni-MWCNT nanoparticles in an epoxy system via TGA, SEM, and OM
- ▣ Fabricate Repair Patch by vacuum resin infusion
- ▣ Evaluate repair patch via TGA, Micro Hardness, and OM



# Carbon Fiber Composites



Reinforcing Phase



Matrix Phase

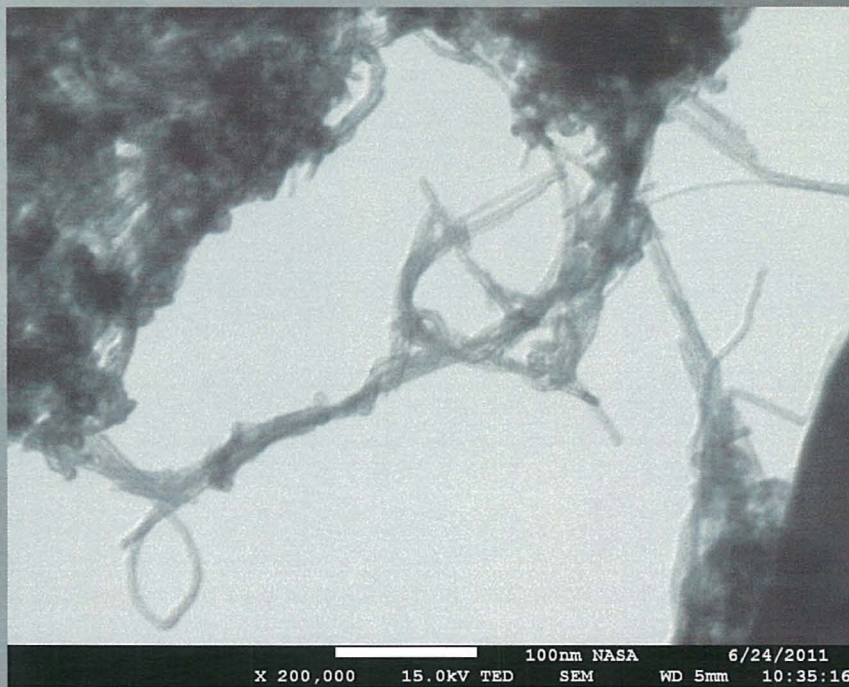


# MWCNT and Ni-MWCNT Nanoparticles

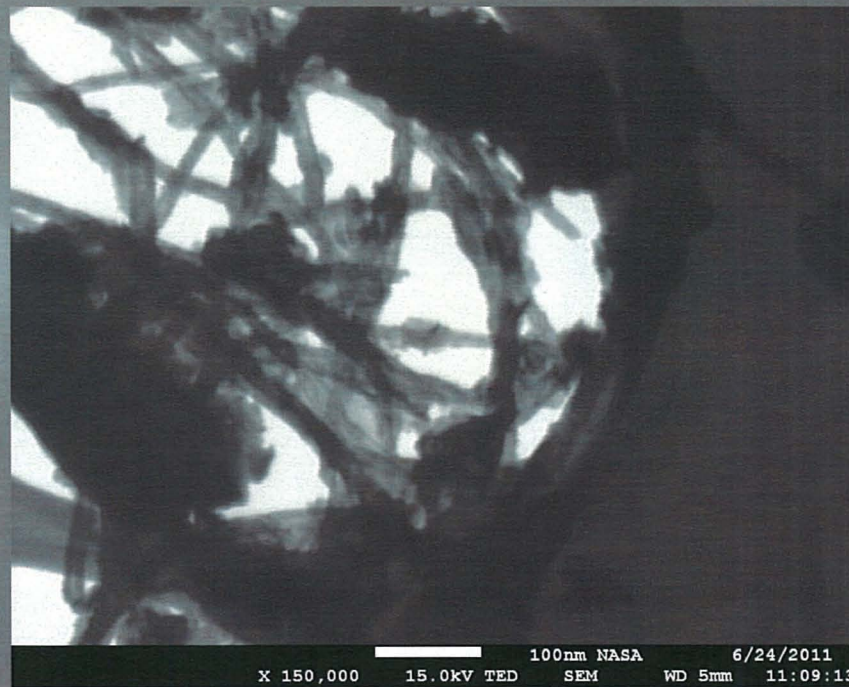
- ▣ Multi-walled carbon nanotubes (MWCNT) are a coaxial assembly of 2 – 50 graphite sheets. Their diameter can range from 5 – 50 nm.
- ▣ Ni can increase conductive properties and electromagnetic shielding of the MWCNTs.



# SEM of MWCNT and Ni-MWCNT



MWCNT

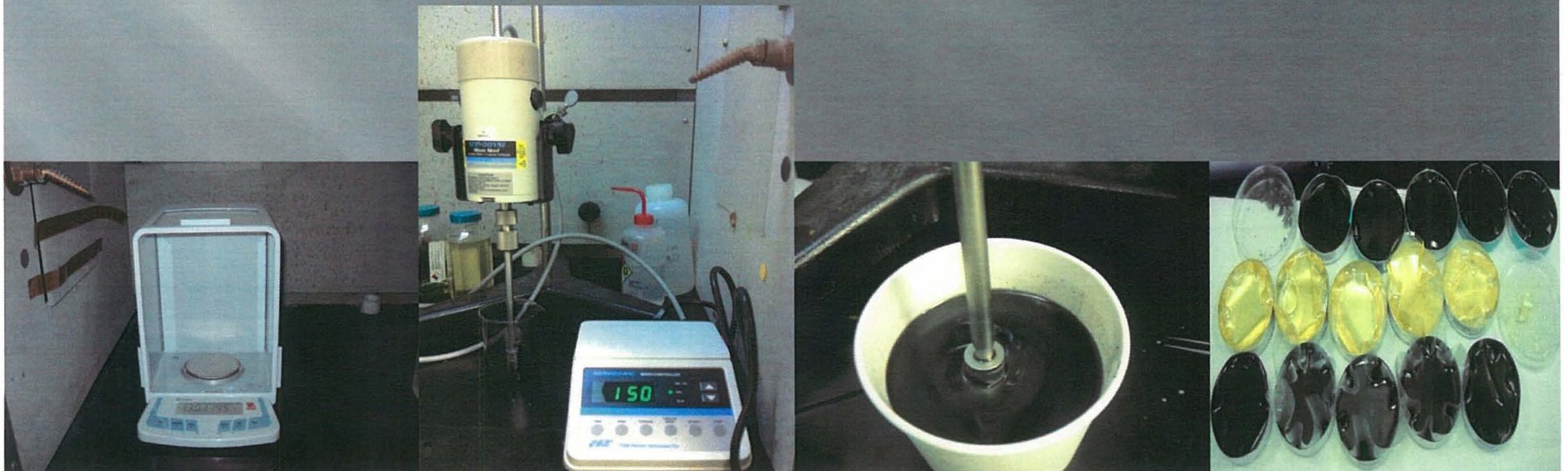


Ni-MWCNT



# MWCNT/Epoxy and Ni-MWCNT/Epoxy Cure: Method A

- ▣ The hardener and carbon nanotubes were weighed on an analytical balance.
- ▣ The carbon nanotubes were added to the hardener and mixed with a Servodyne mixer for 30mins at 150rpm.
- ▣ The carbon nanotubes and hardener were added to the resin in a plastic cup and mixed with the Servodyne mixer for 10mins.
- ▣ The Epoxy system and carbon nanotubes were added to aluminum pans lined with parafilm used as release film.





# SEM Sample Preparation

- ▣ The cured samples were sectioned into pieces with a Struers 36TRE cut off wheel.
- ▣ One of the sample sections was hit with a hammer to obtain shard samples.
- ▣ The pieces were mounted on an SEM stub with carbon tape.
- ▣ The samples were gold-coated to prevent the samples from building up electrons or charging.

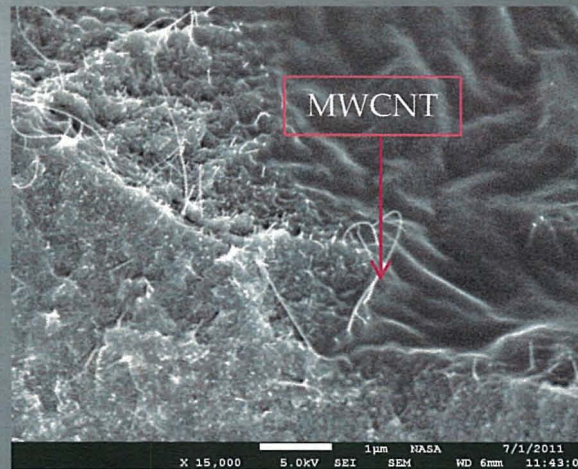




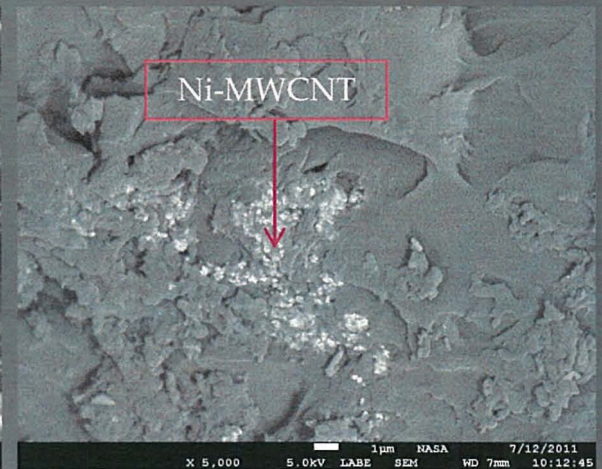
# SEM Images



Neat Epoxy



MWCNT/Epoxy System

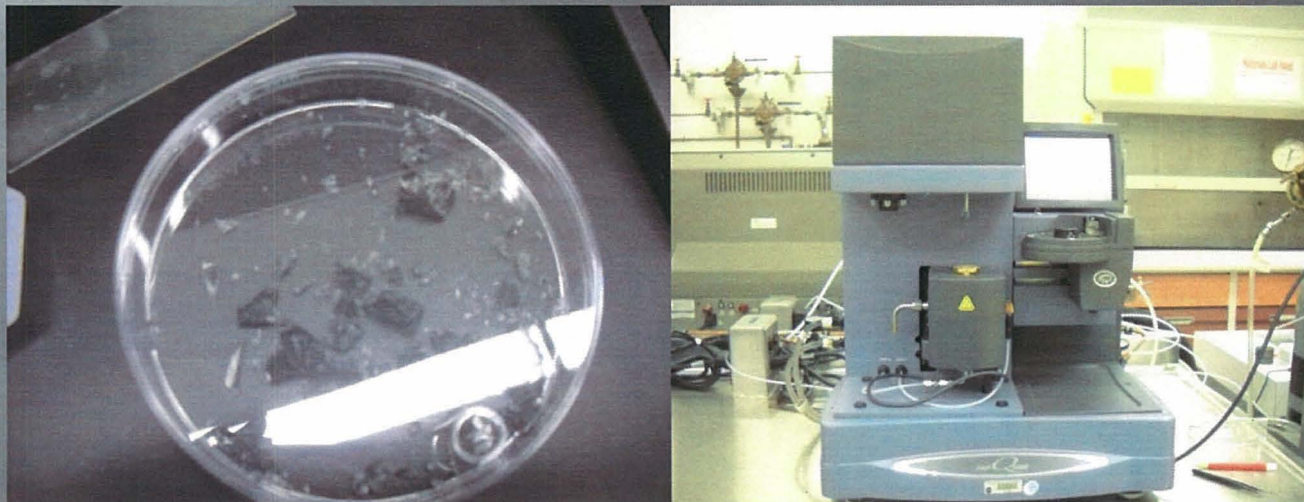


Ni-MWCNT/Epoxy System



# TGA: Method A Samples

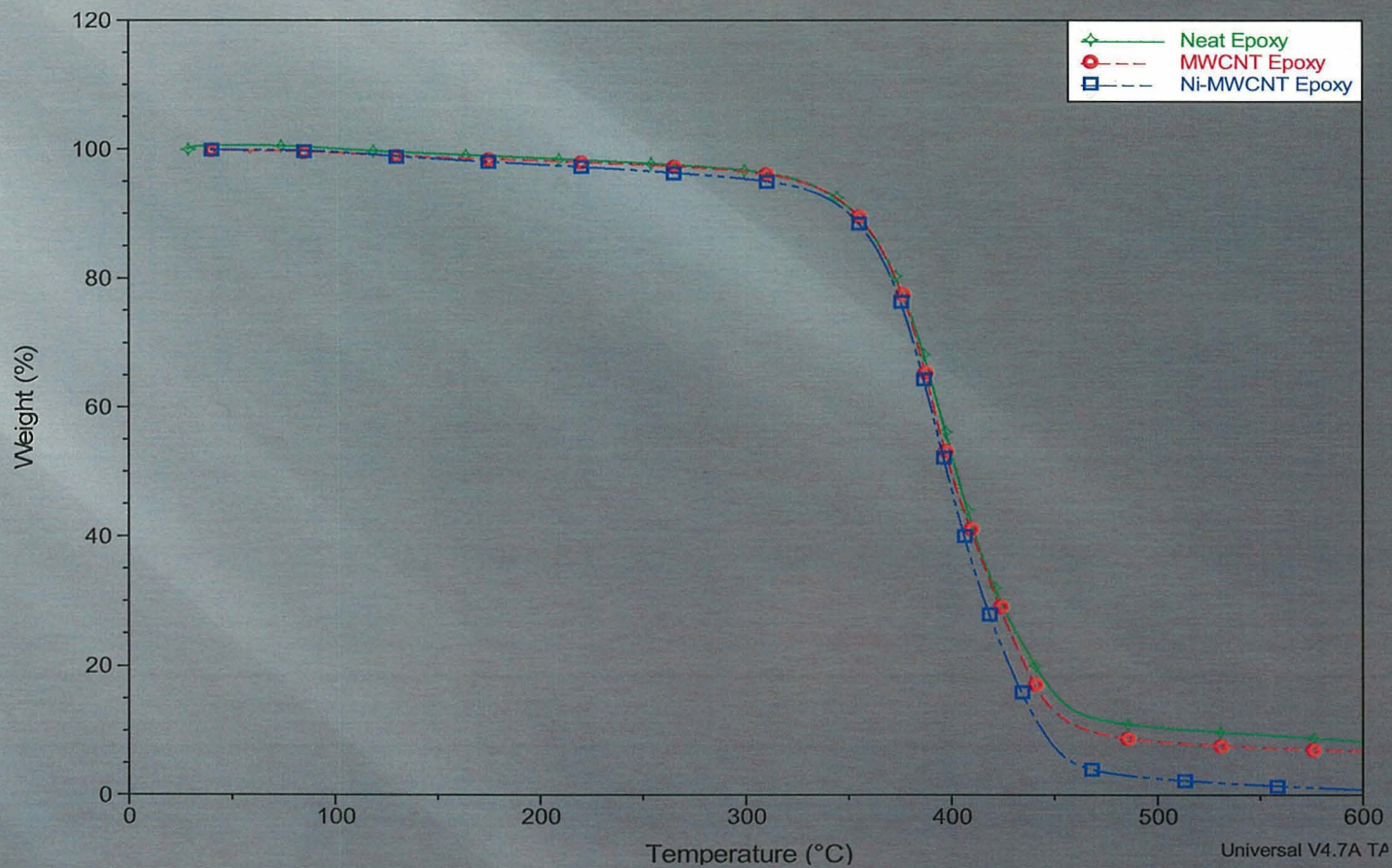
- ▣ Shard samples were run at 20°C/min to 800°C





# TGA Data: Method A

Sample Identification	Temperature (°C)
Control/Neat Epoxy	363
MWCNT/Epoxy System	362
Ni-MWCNT/Epoxy System	363





# MWCNT and Ni-MWCNT Cure in Epoxy: Method B

- ❑ The hardener and carbon nanotubes were weighed with an analytical balance.
- ❑ The carbon nanotubes were mixed with the hardener using a probe sonicator in an ice bath.
- ❑ The hardener mixed with the nanotubes was added to the resin in a plastic cup.
- ❑ The Epoxy system and carbon nanotubes were mixed with a wooden stirrer.





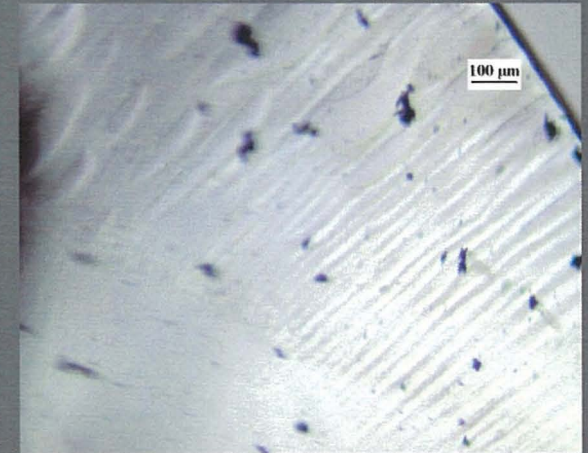
# Optical Microscope Images



Neat Epoxy



MWCNT/Epoxy System



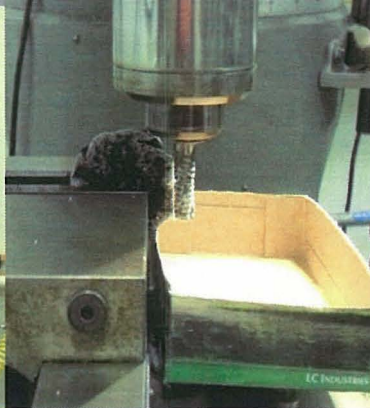
Ni-MWCNT/Epoxy System



# TGA: Method B Samples

1. Small sample shavings were milled from the cured samples.
2. Sample preparation was modified to hitting a piece of the cured samples to acquire shard samples.

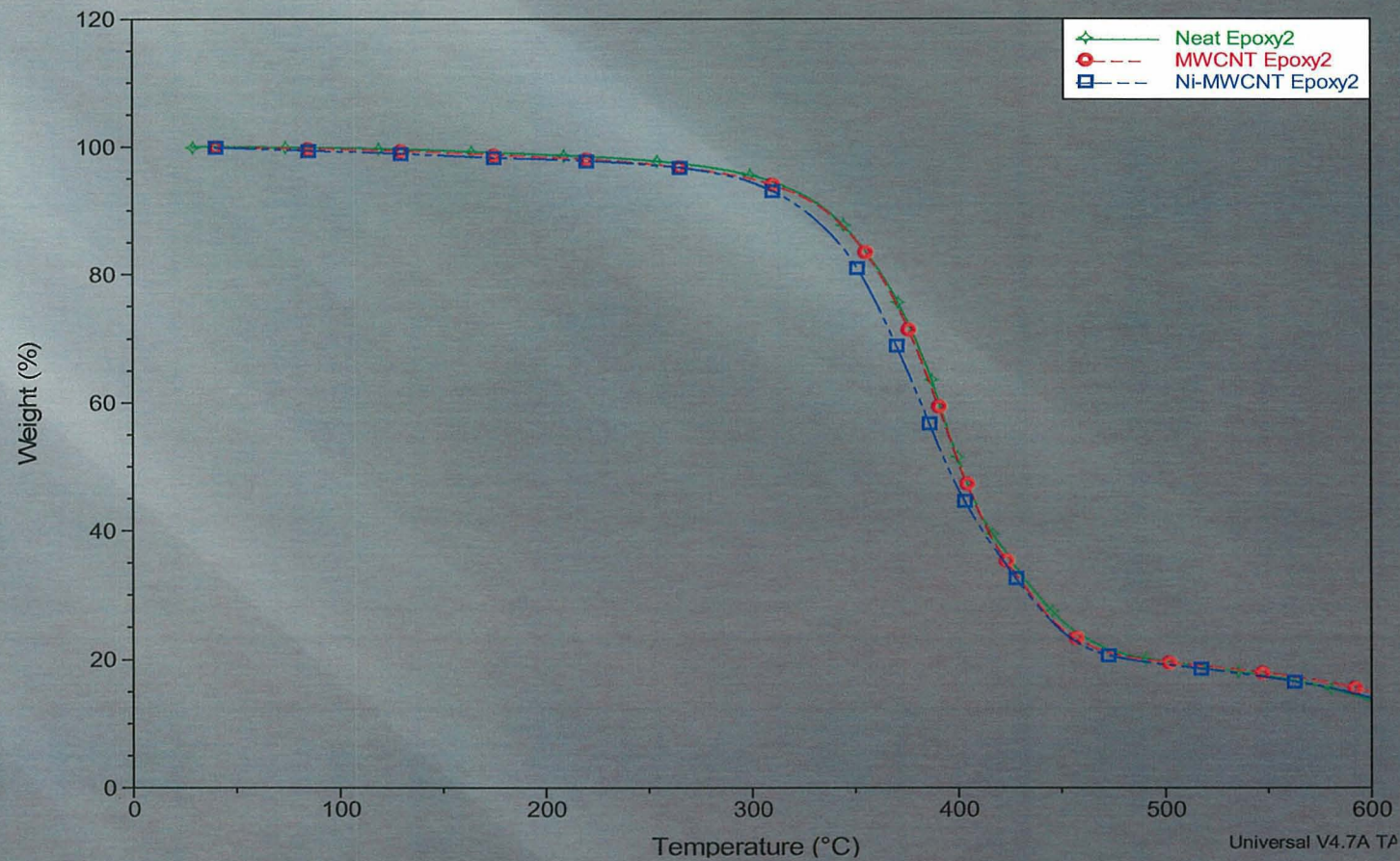
Both samples were run a  $20^{\circ}\text{C}/\text{min}$  to  $600^{\circ}\text{C}$ .





# TGA Method B Sample Shavings

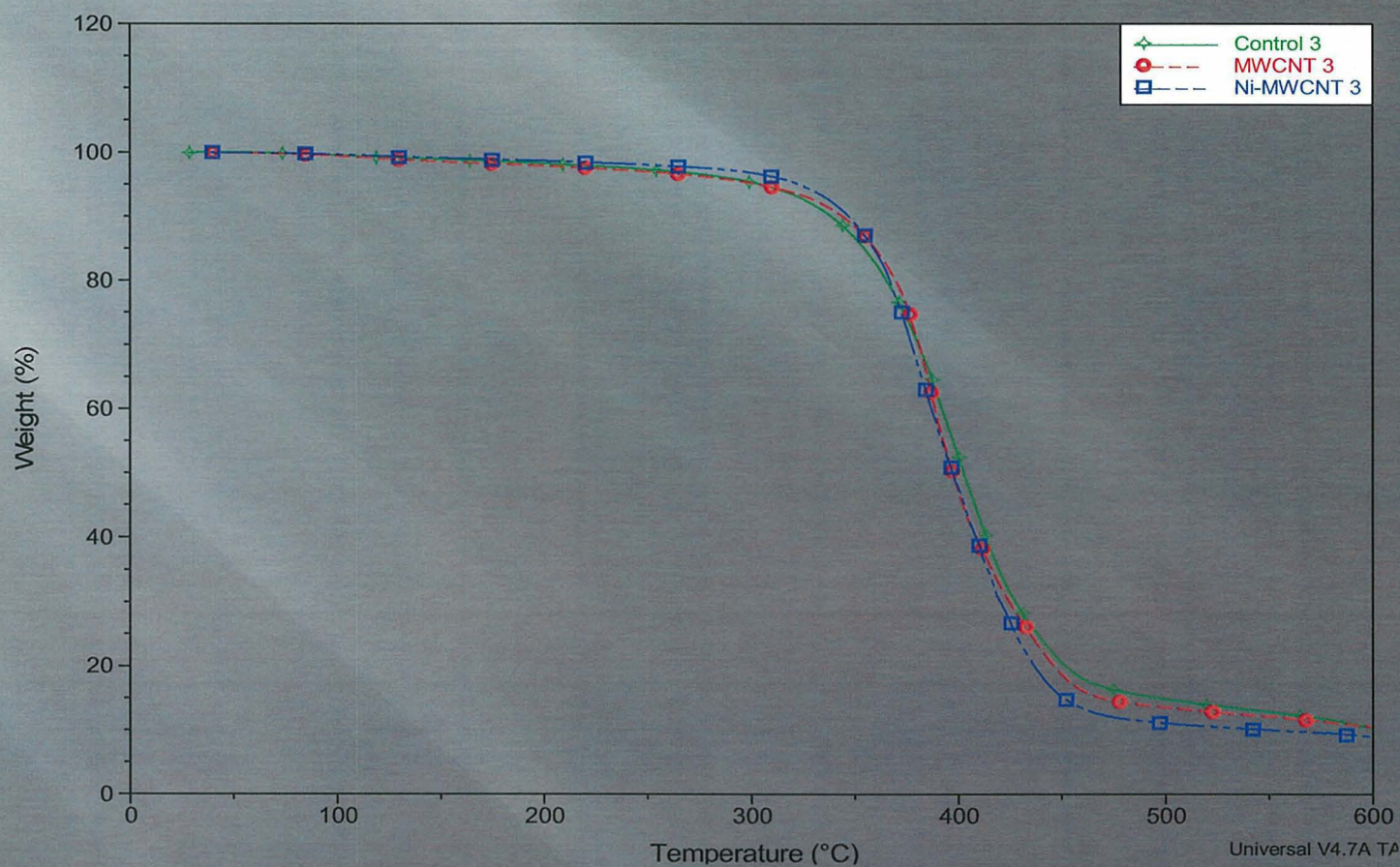
Sample Identification	Temperature (°C)
Control/Neat Epoxy	352
MWCNT/Epoxy System	347
Ni-MWCNT/Epoxy System	334





# TGA Data: Method B Shard Samples

Sample Identification	Temperature (°C)
Control/Neat Epoxy	354
MWCNT/Epoxy System	363
Ni-MWCNT/Epoxy System	352





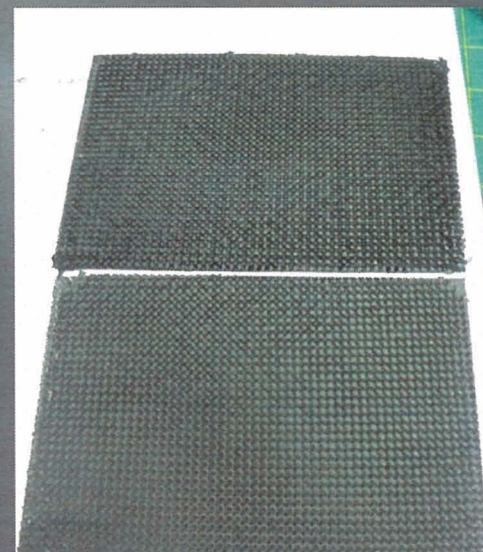
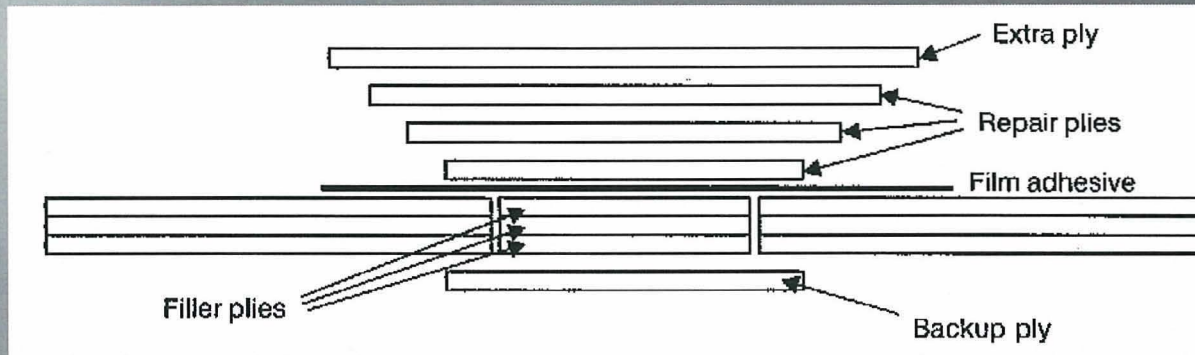
# Infusion Process

- ▣ The infusion lay-up was plastic, carbon fiber patch, peel ply, flow medium, intake and vacuum manifolds, and plastic bag tacky taped to the table.
- ▣ Before infusion, the patch was weighed in the orientation they would be infused.
- ▣ The hardener and carbon nanotubes were mixed using Method B.
- ▣ The hardener and carbon nanotubes were added to the resin and mixed with a wooden stirrer.
- ▣ The Epoxy system and carbon nanotubes were allowed to sit for 10 mins to allow the air bubbles to travel to the surface.
- ▣ The vacuum pump was turn on to pull the lay-up under vacuum.
- ▣ The Epoxy system and carbon nanotubes were infused through a plastic tube and pulled through the carbon fiber by the vacuum pump.
- ▣ When the fibers are fully wet with the epoxy system and carbon nanotubes the plastic tube was clamped closed and the fibers were allowed to cure overnight.
- ▣ The patch was weighed after curing.





# Repair Patch



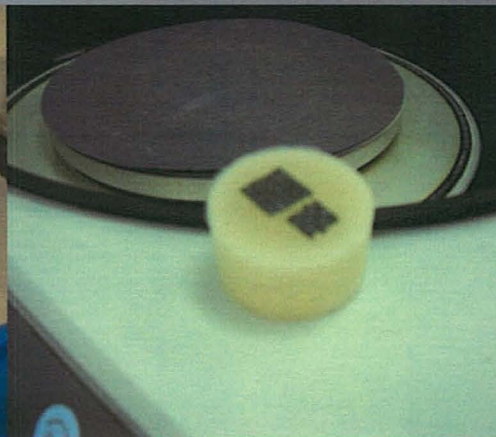
Patch 1:  $[90/90/+45/90]$

Patch 2:  $[0/90/+45/-45]$



# Micro Hardness of Repair Patches

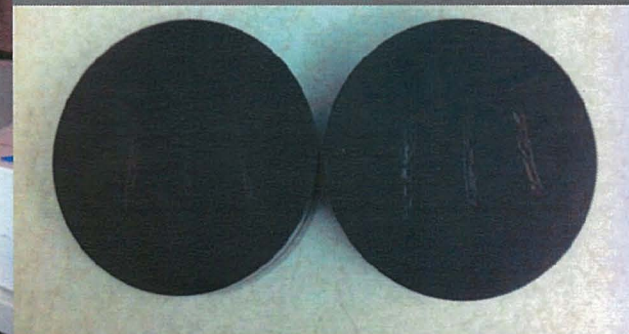
- ❑ Two small squares were cut from each patch.
- ❑ One piece was mounted in Kold Mount specimen mounting material for 30 mins. with the resin rich side on top.
- ❑ The other piece was mounted with the cross section on top.
- ❑ The sample puck was polished to get the excess mounting material off the top.
- ❑ The micro hardness was measured at 50g, 100g, 300g, and 500g loads.





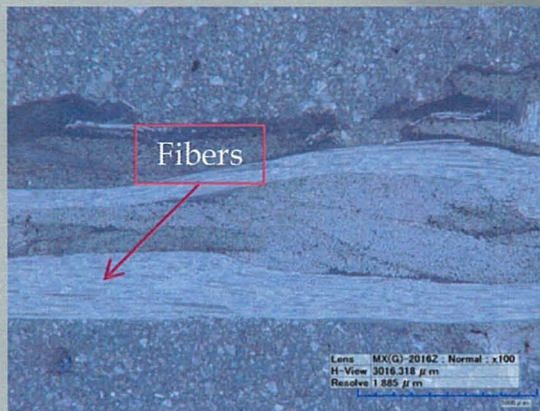
# OM Sample Preparation

- ▣ A small square piece was cut from the patches.
- ▣ The sample put into a multi-clip used for mounting.
- ▣ The samples were mounted in a Phenolic mounting material for 6 mins at 180°C.
- ▣ The sample puck was polished with 120 and 600 grit paper then 9 micron particle paste.

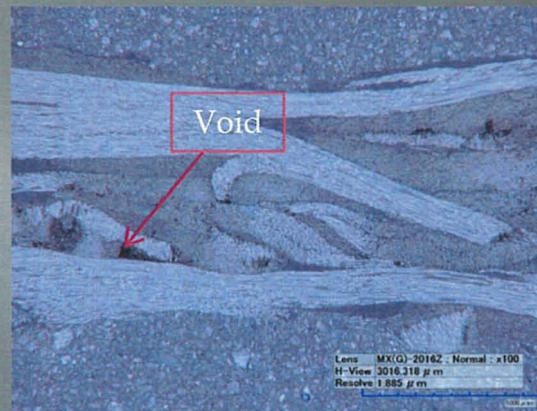




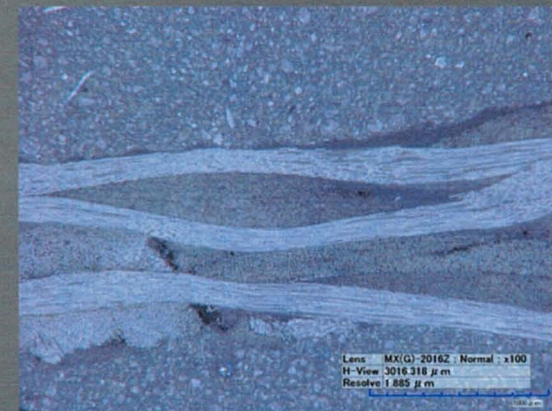
# OM of Repair Patches



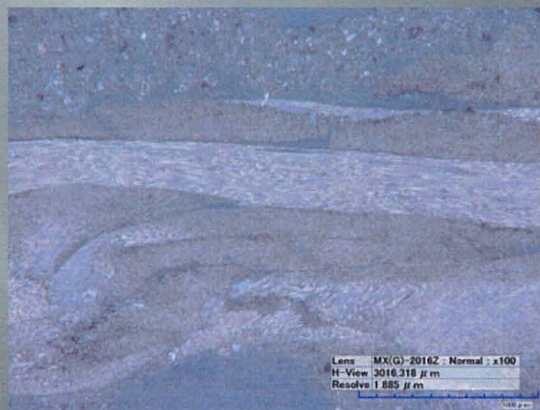
Patch 1: Neat Epoxy in Carbon Fiber



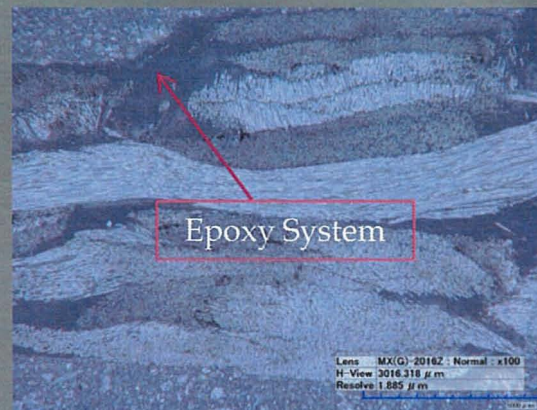
Patch 1: MWCNT/Epoxy System in Carbon Fiber



Patch 1: Ni-MWCNT/Epoxy System in Carbon Fiber



Patch 2: Neat Epoxy in Carbon Fiber



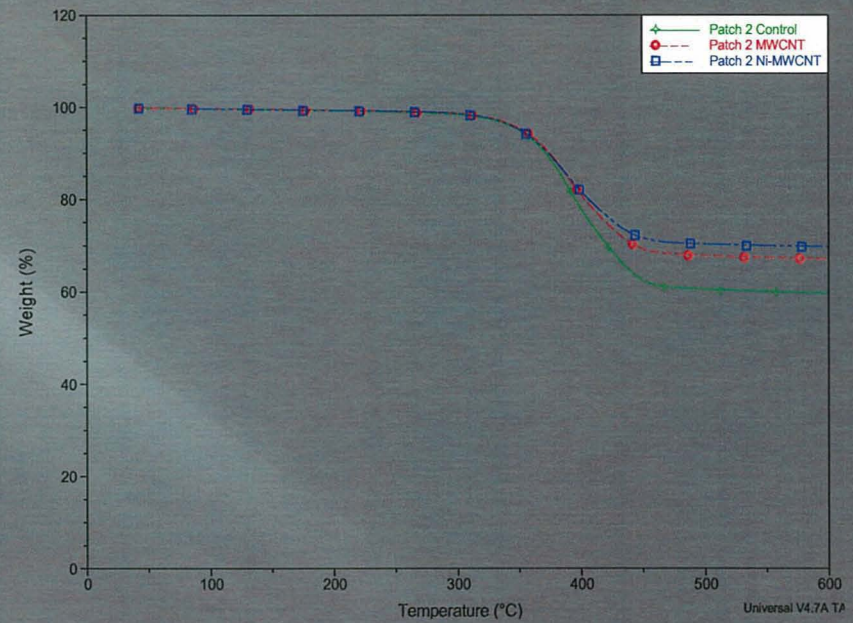
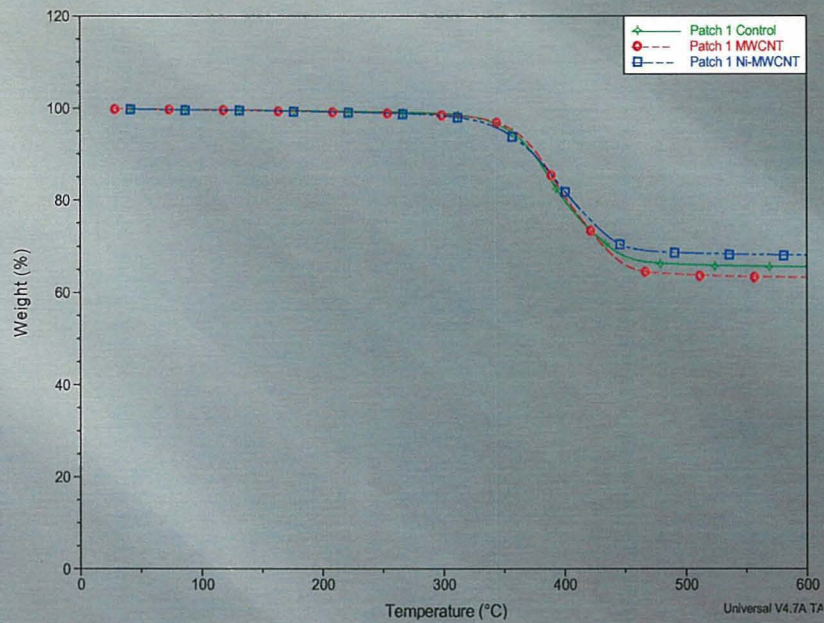
Patch 2: MWCNT/Epoxy System in Carbon Fiber



Patch 2: Ni-MWCNT/Epoxy System in Carbon Fiber



# TGA of Repair Patches



Rate of 20°C/min	Neat Epoxy in CF (°C)	MWCNT/Epoxy System in CF (°C)	Ni-MWCNT/Epoxy System in CF (°C)
Patch 1	354	359	350
Patch 2	354	352	344



# Conclusions

- ▣ Probe sonicator better dispersed the MWCNTs.
- ▣ The Ni-MWCNT were unsuccessfully dispersed with the Sevodyne mixer and probe sonicator.
- ▣ The MWCNT and the Ni-MWCNT did not modify the thermal stability of the epoxy system.
- ▣ The use of the probe sonicator altered the thermal stability by destabilizing the hardener, but the MWCNT stabilized the hardener during sonication.
- ▣ The milling of the MWCNT/epoxy system and Ni-MWCNT/epoxy system also affected the thermal stability.
- ▣ The Ni-MWCNT/epoxy System in carbon fiber had the lowest decomposition temperature for both patches.
- ▣ There were voids and damages in all the repair patches



# Future Work

- ▣ Optimize dispersion technique for Ni-MWCNTs.
- ▣ High-Res TGA.
- ▣ Implement cutting method to decrease repair patch damage.
- ▣ Decrease voids in repair patch.
- ▣ Obtain better understanding of repair patches.



# Acknowledgements

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